

# **STUDY GUIDE**

## **LABORATORY**

### **INTRODUCTION AND ADVANCED**

#### **SUBCLASS J**

WISCONSIN DEPARTMENT OF NATURAL RESOURCES  
BUREAU OF INTEGRATED SCIENCE SERVICES  
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## **PREFACE**

This operator's study guide represents the results of an Ambitious program. Operators of wastewater facilities, regulators, educators and local officials, jointly prepared the objectives and exam questions for this subgrade.

The objectives in this study guide have been organized into modules, and within each module they are grouped by major concepts.

### **HOW TO USE THESE OBJECTIVES WITH REFERENCES**

In preparation for the exams, you should:

1. Read all the objectives that apply to the grade level desired and write down the answers to the objectives that readily come to mind.
2. Use the references at the end of the study guide to look-up answers you don't know. This one set of references covers all of the objectives.
3. Write down the answers found in the references to those objectives you could not answer from memory.
4. Review all answered objectives until you can answer each from memory.

**IT IS ADVISABLE THAT YOU ATTEND SOME FORM OF FORMAL TRAINING IN THIS PROCESS BEFORE ATTEMPTING THE CERTIFICATION EXAM.**

### **Choosing A Test Date**

Before you choose a test date, consider the training opportunities available in your area. A listing of training opportunities and exam dates can be found in the annual DNR "Certified Operator," or by contacting your DNR District operator certification coordinator.

# INTRODUCTION

## INTRODUCTION TO ON-SITE LABORATORY TESTING

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### MODULE A: SAFETY AND LABORATORY EQUIPMENT

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#### CONCEPT: SAFETY

1. Describe the general safety rules governing Laboratory operator behavior for the following:
2. List the personal safety equipment necessary for the safe operation of a wastewater treatment plant Laboratory.
3. Discuss the fire protection equipment necessary for the safe operation of a wastewater treatment plant Laboratory.
4. Describe how to safely perform each of the following Laboratory tasks:
  - A. Smelling chemicals.
  - B. Mixing acid and water.
  - C. Making glass-to-rubber connections.
  - D. Using a Pipet.
5. Discuss safe storage of Laboratory chemicals.

#### CONCEPT: LABORATORY EQUIPMENT

6. List the equipment and glassware commonly found in a wastewater treatment plant Laboratory.
7. Explain the function of an incubator in a wastewater treatment plant Laboratory.
8. Explain the function of a desiccator in a wastewater treatment plant Laboratory.
9. List the operating precautions that will help maintain the precision and accuracy of an analytical balance.

10. Discuss the glass and equipment cleaning procedures after tests for the following:

- A. BOD
- B. Total Suspended Solids(T.S.S.)

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**MODULE B: OPERATION AND CHEMICALS**

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**CONCEPT: OPERATION**

11. State the operating temperature requirements in °C for each of the following:

- A. BOD Incubator.
- B. Suspended Solids Drying Oven.
- C. Refrigerator.
- D. Composite Sampler.
- E. Muffle Furnace.

12. Explain how each of the following are used in a wastewater treatment plant Laboratory:

- A. Volumetric Pipet.
- B. Measuring Pipet.
- C. Buret.
- D. Microburette.
- E. Volumetric Flask.

13. Describe the size and type of pipets used in BOD and TSS tests.

14. Identify the causes and corrective action to eliminate air bubbles or liquid clinging to the side of a buret or pipet.

15. Explain the proper way to read a water level meniscus.

16. Define the term and explain why filter and sample residue should be dried to a "constant weight."

17. Describe the color and consistency of "old" non-indicating and indicating desiccant.

18. List the conditions that may make it difficult for a drying oven to maintain its temperature.
19. Explain what data must be recorded when receiving new chemicals at a Laboratory.
20. Define the following terms:
  - A. Arithmetic Mean.
  - B. Range.
21. Explain the following units commonly used in recording Laboratory data:
  - A. mg/L.
  - B. S.U.
  - C. MG/KG.
  - D. % Solids.
  - E. N
  - F. UMHOS/CM.
22. Describe the characteristics of a good Bench Sheet.
23. Describe the proper means of record filing and the length of retention of completed Bench Sheets or Laboratory data.
24. Given flow, calculate pounds of BOD and Suspended Solids per day.

**CONCEPT:   CHEMICALS**

25. Identify and explain the use of the following two grades of chemicals used in a wastewater Laboratory.
  - A. Technical Grade.
  - B. Analytical Reagent Grade.

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**MODULE C: LABORATORY WATER ANALYSIS AND SAMPLING**

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**CONCEPT: LABORATORY WATER ANALYSIS**

26. Discuss how to handle the first few liters of water from a distillation or deionization unit.
27. List the most likely Laboratory water contaminants and their sources.
28. Discuss the term "Laboratory Grade" and the proper storage of Laboratory pure water.
29. Describe what is measured in laboratory water when it is tested for conductivity.

**CONCEPT: SAMPLING**

30. Describe the cleaning and preparation of the following types of sample containers and devices.
  - A. General Sample Containers.
  - B. Manual Composite Devices.
  - C. Stationary Composite Devices.
  - D. Flow Through Devices.
  - E. Portable Devices.
31. Define the following terms:
  - A. Duplicate Sample.
  - B. Aliquot Sample.
  - C. Reagent Blank.
  - D. Trip Blank.
  - E. Sample Blank.
32. Describe the guidelines to consider prior to collecting a representative sample.
33. Explain the difference between grab and composite samples.

34. Discuss how to determine if contaminants are being introduced in the sampling process.
35. List the information to be included in a sample log, and on the sample container.
36. Identify the maximum holding times and preservation methods for the following samples:
  - A. BOD
  - B. Total Suspended Solids (T.S.S.).
  - C. Ammonia ( $\text{NH}_3$ ).
  - D. Total Phosphorus
  - E. Fecal Coliform
37. Discuss the action a laboratory must take when given a warning or control limit.

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#### **MODULE D: LABORATORY ANALYSIS**

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##### **CONCEPT: pH ANALYSIS**

38. Describe how pH affects wastewater treatment and how Ph units are reported.
39. Discuss the procedure for pH measurement, and the calibration of a pH meter.

##### **CONCEPT: DISSOLVED OXYGEN (D.O.) ANALYSIS**

40. Describe why some wastewater treatment plants are required to maintain minimum D.O. in their effluent, while others do not.
41. Explain what is measured in a D.O. analysis.
42. Discuss when the D.O. test should be run.



43. Explain the relationship between temperature and dissolved oxygen for the following:
  - A. Wastewater Treatment Plant Operation.
  - B. When Conducting a D.O. Analysis.
44. Describe the following methods of measuring D.O.:
  - A. Standard Membrane Electrode System and Meter.
  - B. Winkler Titration (An Iodometric Titration).
45. Outline how to calibrate a D.O. meter.
46. List some causes for inaccurate D.O. readings when performing the following:
  - A. If the Winkler Titration Method is being used.
  - B. If a D.O. Meter is being used.
47. Identify the reasonable limits of accuracy and precision for a properly operating D.O. meter.
48. List items to consider when inspecting a standard membrane electrode D.O. probe for wear.

**CONCEPT: BIOCHEMICAL OXYGEN DEMAND(BOD) ANALYSIS**

49. Define Biochemical Oxygen Demand(BOD).
50. Identify acceptable sample D.O. depletions in a BOD analysis.
51. Define the following analysis, and give an example of an appropriate use of each:
  - A. Total BOD.
  - B. Carbonaceous BOD (CBOD).
  - C. Soluble BOD.
  - D. Nitrogenous BOD (NBOD).
  - E. Chemical Oxygen Demand (COD).
52. List the chemicals used in the preparation of BOD dilution water, and explain their purpose.
53. Describe how to prepare and store BOD dilution water.
54. Outline the BOD analysis procedure.

55. Define and give examples of the toxic effects or interferences in BOD analysis.
56. Given data, calculate the mg/L BOD.
57. Explain what to do if a sample is overdepleted and underdepleted.

**CONCEPT: TOTAL SUSPENDED SOLIDS (T.S.S.) ANALYSIS**

58. Identify the types of solids included in a T.S.S. Analysis.
59. Identify the filter type used in T.S.S. analysis.
60. Explain how filters should be prepared before being used in T.S.S. analysis.
61. Describe how to mix a T.S.S. sample prior to filtration.
62. Outline the T.S.S. analysis procedure.
63. Given data, calculate the Total Suspended Solids concentration.
64. Explain why a desiccator should seal properly during the T.S.S. analysis.

**CONCEPT: LABORATORY QUALITY ASSURANCE**

65. Define Laboratory Quality Assurance (QA).
66. Explain the advantages of an interlab testing program.
67. Discuss the considerations that might lead a laboratory to utilize contract laboratories.

**ADVANCED**

## ADVANCED ON-SITE LABORATORY TESTING

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### MODULE A: LABORATORY EQUIPMENT AND OPERATION

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#### CONCEPT: LABORATORY EQUIPMENT

1. Explain why and how laboratory thermometers are calibrated.
2. Describe the spectrophotometer and it's operating procedure.
3. Explain how to accurately check the calibration on an analytical balance.

#### CONCEPT: OPERATION

4. Discuss the tests in which spectrophotometers are commonly used.
  5. Discuss what effect a low or high oven temperature will have on the sample results.
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### MODULE B: CHEMICALS AND LABORATORY WATER ANALYSIS

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#### CONCEPT: CHEMICALS

6. Calculate how much water and how much chemical should be used to obtain a specific normality when using a dry chemical reagent.
7. Calculate the volume of a 1.0N solution needed to make 1 liter of a 0.025N solution.
8. Explain the procedure to standardize sodium thiosulfate (thio).

**CONCEPT:    LABORATORY WATER ANALYSIS**

9. List the quality control tests to be conducted on laboratory water.
  10. Describe how to run a potassium permanganate ( $\text{KMnO}_4$ ) test and interpret the results.
  11. Identify the laboratory tests affected if the following problems occur with the laboratory distilled water:
    - A. High Copper or Chrome levels.
    - B. Dissolved Biodegradable Solids.
    - C. Excess Conductivity.
  12. List the sources of oxygen demand in dilution water.
  13. Explain resistivity and conductivity as they relate to the grades of laboratory water.
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**MODULE C: TROUBLESHOOTING AND SAMPLING**

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**CONCEPT:    TROUBLESHOOTING**

14. Explain what can happen if a sample, plus dilution water, is significantly colder than  $20^\circ\text{C}$ . before being placed in the incubator.
15. Discuss the effect on the results of a BOD analysis if the incubator temperature was changed to  $22^\circ\text{C}$  during the analysis.
16. Outline a troubleshooting procedure to determine the cause of an apparent blank depletion.
17. List the possible causes of INCREASED dissolved oxygen in a dilution water blank.
18. Describe how a partially nitrified sample may affect the BOD analysis results.

**CONCEPT: SAMPLING**

19. Define how each of the following are measured:
  - A. Total Kjeldahl Nitrogen.
  - B. Ammonia Nitrogen.
  - C. Nitrate Plus Nitrite Nitrogen.
20. Explain how to make serial dilutions of a sample.
21. Explain the items to consider in preparing a portable sampler for collection system monitoring.
22. Explain why the filter-plus-sample should be dried to a constant weight.
23. Explain how the following sampling errors might alter laboratory results:
  - A. Poor Sampling Location.
  - B. Improper Use of Grab Vs. Composite Sampling.
  - C. Sampling Equipment Not Properly Cleaned.
  - D. Composite Sampler Not Cold Enough For Storage.
  - E. Improper or Lack of Chemical Preservation.
  - F. Composite Sampler Not Set Properly.
24. Given data, calculate the concentration of spike added to a sample.
25. Given a spiked sample analysis, calculate its percent recovery.

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**MODULE D: LABORATORY ANALYSIS**

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**CONCEPT: pH ANALYSIS**

26. Describe how to inspect, predict useful life, and store a Ph electrode.
27. Explain why the fill hole on a reference electrode should be open when testing for pH, and closed when the probe is not in use.

28. Discuss the advantages and disadvantages of a gel-filled pH electrode.
29. Identify the possible causes and corrective actions for the following pH meter problems:
- A. The Instrument Does Not Give A Reading.
  - B. The Two-Point Calibration Differs By More Than .1 pH Unit.
  - C. There Is A Slow Response Time.
  - D. The Needle Will Not Stabilize.
  - E. The Reference Junction Is Plugged.
  - F. There Is A Crystal Formation Inside The Probe.
30. Describe the function of a shorting strap on a pH meter.

**CONCEPT: DISSOLVED OXYGEN (D.O) ANALYSIS**

31. Identify the possible causes and corrective actions for the following Dissolved Oxygen (D.O.) meter problems:
- A. The Meter Will Not Redline.
  - B. The Meter Is Slow To Reach Endpoint.
  - C. There Is A Darkened Gold Electrode.
  - D. There Is A Darkened Silver Electrode.
32. Describe the Winkler Dissolved Oxygen procedure.
33. Identify the reasonable limits of accuracy and precision for a properly operating Winkler titration.

**CONCEPT: BIOCHEMICAL OXYGEN DEMAND (BOD) ANALYSIS**

34. Outline the acceptable seeding procedures for a BOD reference sample.
35. Explain where seed for the BOD analysis should be collected, and how it should be handled.
36. Given data, calculate BOD of a seeded sample.
37. Discuss the process used to dechlorinate the final effluent for a BOD test.
38. Discuss how to select appropriate dilutions based on approximate BOD values.

39. Given bench sheet data, determine which aliquot's values should be accepted and which should be averaged if more than one value is acceptable.
40. Describe how to determine if there is a potential problem with sample toxicity during a BOD analysis.
41. Discuss a procedure to identify the cause of the following problems in the BOD test:
  - A. Toxic Dilution Water.
  - B. Organic Contaminants In Dilution Water.

**CONCEPT: SOLIDS ANALYSIS (TOTAL AND SUSPENDED)**

42. List the purposes of running a blank filter through the suspended solids analysis procedure along with the samples.
43. Explain why it is recommended to heat a total solids sample on a steam bath until it appears dry before putting it in a drying oven.
44. Discuss the procedure for total and volatile solids, determination for sludge, and give an examples where its use would be appropriate.

**CONCEPT: LABORATORY QUALITY ASSURANCE(QA)**

45. Define the following as they relate to laboratory quality assurance:
  - A. Precision.
  - B. Accuracy.
  - C. Quality Control Analysis.
  - D. Blind Standards.
  - E. Reference Standards.
  - F. Spiked Samples.
  - G. Range.
  - H. Significant Figures.
  - I. Outlier.
46. Describe the techniques laboratory analysts use to determine accuracy and precision of data.
47. List the elements of a Quality Assurance program.



48. Explain how an acceptable performance for a reference sample is determined, and discuss actions to be taken if a plant fails a reference sample test.
49. Look at a series of sample control charts and be able to state:
  - A. Which results are in control.
  - B. What actions or remedial steps are required of the operator in problem situations.

**CONCEPT: LABORATORY QUALITY CONTROL(QC)**

50. Describe how to find the average range, set a warning limit, and a control limit for quality control.
51. Explain how to calculate the following:
  - A. Spike Added Concentration.
  - B. Percent Recovery.
  - C. Standard Deviation of a Recovery.
  - D. Control and Warning Limits for QC.
52. Explain which results should be excluded from a data base when computing control limits.
53. Explain why re-evaluation of control chart limits may be necessary more or less often than once every six months as suggested in the EPA Quality Control manual.

## RESOURCES

1. A USER'S GUIDE TO LABORATORY SERVICES. (1989). Arneson, Ronald. Wisconsin Department of Natural Resources, Office of Technical Services, P.O. Box 7921, Madison , WI 53707.
2. CONTROLLING WASTEWATER TREATMENT PROCESSES. (1984). Cortinovis, Dan. Ridgeline Press, 1136 Orchard Road, Lafayette, CA 94549.
3. HANDBOOK FOR SAMPLING AND SAMPLE PRESERVATION OF WATER AND WASTEWATER. EPA-600/4-82-029 (1982). U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.
4. LABORATORY SAFETY MANUAL. Mallinckrodt Chemical Works, Science Products Division, St.Louis, MO 63160.
5. OPERATION OF MUNICIPAL WASTEWATER TREATMENT PLANTS. Manual of Practice No.11(MOP 11), 2nd Addition (1990), Volumes I,II,andIII. Water Environment Federation (Old WPCF), 601 Wythe Street, Alexandria, VA 22314-1994. Phone (800) 666-0206.
6. OPERATION OF WASTEWATER TREATMENT PLANTS. 3rd Edition (1990), Volumes 1 and 2, Kenneth D. Kerri, California State University, 6000 J Street, Sacramento, CA 95819-6025. Phone (916) 278-6142.
7. OPERATION OF WASTEWATER TREATMENT PLANTS. Manual of Practice No.11 (MOP 11)(1976). Water Pollution Control Federation, 601 Wythe Street, Alexandria, VA 22314-1994. Phone (800) 666-0206. (Probably Out-Of-Print, See Reference Number 5).
8. QUALITY ASSURANCE DOCUMENT FOR A SMALL WASTEWATER LAB. 2<sup>nd</sup> Edition (1992). Department of Natural Resources, Office of Technical Services, P.O. Box 7921, Madison, WI 53707.
9. SIMPLIFIED LABORATORY PROCEDURES FOR WASTEWATER EXAMINATION. (1985). Water Environment Federation (Old WPCF), 601 Wythe Street, Alexandria, VA 22314-1994, Phone (800) 666-0206.
10. STANDARD METHODS FOR THE EXAMINATION OF WATER AND WASTEWATER. 17th Edition (1989), 18th Edition (1992). Joint Publication of: American Public Health Association; American Water Works Association; and, Water Environment Federation (Old WPCF). Publication Office: American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005.

11. **WISCONSIN ADMINISTRATIVE CODE, NR 149, LABORATORY CERTIFICATION AND REGISTRATION.** Wisconsin Department of Natural Resources, Attn: Ken Cramer, P.O. Box 7921, Madison, WI 53707.
12. **WISCONSIN ADMINISTRATIVE CODE, NR 218, METHOD AND MANNER OF SAMPLING.** Wisconsin Department of Natural Resources, Attn: Ken Cramer, P.O. Box 7921, Madison, WI 53707.
13. **WISCONSIN ADMINISTRATIVE CODE, NR 219, ANALYTICAL TEST METHODS AND PROCEDURES.** Wisconsin Department of Natural Resources, Attn: Ken Cramer, P.O. Box 7921, Madison, WI 53707.